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George K. Burgess, Director

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AS A PAPER-MAKING MATERIAL**

By Merle B. Shaw and George W. Bicking

TECHNOLOGIC PAPERS OF THE BUREAU OF STANDARDS, No. 340

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BY

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Bureau of Standards

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ABSTRACT

Experimental tests made at the bureau to determine the paper-making quality of caroá fiber have shown that material to be very satisfactory for paper manufacture on the basis of both quality of pulp produced and yield obtained. The tests have indicated that the fiber is especially suitable for use with or as a substitute for rag and rope stock, of which there is a scarcity in the paper industry.

Caroá is a plant of the pineapple family and is indigenous to eastern Brazil. By beating the leaf between stones the natives separate the fiber, which is used locally for coarse hand-made twines, nets, and rope. Caroá is not employed commercially, however, but production and cost estimates indicate that its use for paper making would be practical.

Three samples of caroá, each having received different treatment in the separation of the fiber previous to shipment, were employed in the paper-making tests. The caustic soda process was used in the preparation of the pulp. With small amounts of caustic the paper produced was very strong and well suited for bag or wrapping purposes. With larger amounts the pulp bleached easily to a good white, and the paper made compared favorably with papers made from rag stock. The chemical consumption and yield of pulp were satisfactory.

A micrographic study showed the fiber to be comparatively long and of small diameter. These properties tend to produce good felting of the fibers and give compactness and strength to the resulting sheet.

Comparative test data on kraft, sulphite, and rag pulps are included in the publication. The laboratory paper-making equipment and technic employed in paper research at the bureau also are described in detail.

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I. INTRODUCTION

Until about half a century ago practically the only raw material for commercial paper making consisted of rags and waste cordage. The available quantity of these materials was inadequate, however, to meet the increasing demand for paper products. The development of the processes of making pulp from wood resulted, therefore, in that substance being drawn upon almost entirely in the expansion of the paper industry. The fiber content of many grades of papers, such as book, newsprint, and the cheaper writings, is now composed exclusively of wood pulp. For fine papers in which unusual strength is required—ledgers, bonds, currency—rags are still used as formerly. For strong bag or wrapping paper, in which strength is more important than color, worn and salvaged rags, old rope, and waste tow are generally utilized. There is also, however, a scarcity of rag and rope stock for these latter uses, and the problem of finding new fibers possessing the essential characteristics of these materials is receiving attention. The investigation herein reported demonstrates the suitability of caroá fiber for use with or as a substitute for rag and rope stock in paper production.

II. OCCURRENCE AND CHARACTERISTICS OF CAROÁ

1. GENERAL¹

Caroá (*Neoglaziovia variegata*), variously spelled carua, caroa, caroá, is a plant of the pineapple family (*Bromeliaceæ*). It is indigenous to eastern Brazil, being especially abundant in the valley of the Sao Francisco River, and grows under somewhat arid conditions at such altitudes as become rather cold in winter. Its thickness of growth and reproducibility after harvesting have made cultivation unnecessary for supplying the needs of the natives. If, however, extensive supply were required for use on a large commercial scale, cultivation would doubtless become important.

The leaf of the caroá plant is about 4 feet long and from 1½ to 2 inches wide, being slightly larger than the leaf of the pineapple, which in certain parts of the world, notably the Philippine Islands, is extensively used for the manufacture of textiles. By beating the leaf between stones the natives roughly separate the fiber, which after subsequent retting and washing is fashioned into coarse hand-made twines for use locally in nets, fishing lines, and rope.

Caroá rope is used for binding hides and skins into bales for shipment and tying loads on pack animals for transportation from the interior to the seacoast, but it is not a material of commerce. It is

¹ The authors are indebted to L. H. Dewey, botanist in charge of fiber investigations, Bureau of Plant Industry, Washington, D. C., for botanical information included; and to the Bureau of Foreign and Domestic Commerce, Washington, D. C., for production and economic data received from the American consulate, Pernambuco, Brazil.

made on the farms from which shipments leave because it is cheaper than rope purchased and delivered to the interior. Caroá fiber, although strong and resistant, is unattractive for cordage as compared with abaca, sisal, and other hard fibers now used for this purpose. It is not recommended for the manufacture of cordage because the fibers are too hard and do not clean easily. The pulpy substance which remains after cleaning is subject to rapid fermentation and decomposition in the presence of moisture.²

Although there is no organized caroá industry, it is believed that, if there were sufficient demand for the fiber to make harvesting and transporting commercially profitable, a steady supply of considerable quantity could be obtained if a permanent market were assured. The cost at which it is estimated that it could be imported in quantity³ would permit the use of caroá for paper-making purposes. Before extensive supply of the fiber could be procured, however, development of methods of production and preliminary separation of the fiber might materially affect the present price. It is reported that, previous to the separation of the fiber, other materials (gum, oil, balsam, pitch, and acid) that may be of even greater value can be extracted.

2. TEST SAMPLES

Three different samples were employed in the laboratory paper-making tests. The preliminary treatment and source of each were stated by those submitting the material as follows:⁴

A. Shredded only; source, Caruarú, Pernambuco; obtained and forwarded by the American Consul, Pernambuco, Brazil.

B. “* * * is the ‘batido’ (beaten) which is cut and beaten without immersion in water”; source, Bahia, Brazil; supplied by an American commercial house.

P. “* * * is the ‘pubo’ (fermented) which is obtained by soaking the cut stalks in water for a period of about 10 days, after which the gum is extracted”; source, Bahia, Brazil; supplied by an American commercial house.

The caroá supplied for the tests conducted on a semicommercial scale was similar to sample “B.”

III. LABORATORY PAPER-MAKING TESTS

Inasmuch as the equipment and procedure employed in the preparation of the caroá pulp and its subsequent conversion into paper are those used at the bureau for the laboratory paper-making tests

² “Industria de Fibras,” J. Reynal, Bulletin of the Ministry of Agriculture, Industry and Commerce, October-November, 1922, pp. 125-136; Rio de Janeiro.

³ Seven to ten cents per pound, f. o. b. New York, has been quoted for treated material.

⁴ Acknowledgment for samples B and P and the material for the semicommercial tests is due Adolph Hirsch & Co (Inc.), New York, N. Y.

of all materials under investigation, detailed descriptions are given. These may be of interest and value to those engaged in similar paper research.

1. EQUIPMENT

(a) **BOILER.**—In the process of separating the cellulose, or fiber, from the accompanying noncellulose substances (which are undesirable in paper and must be eliminated) a small cylindrical rotary boiler is employed. This boiler, shown in Figure 1, has been found to give excellent results in laboratory-scale pulping experiments. Its length is 18 inches, diameter 8 inches, and capacity 12.5 liters. It is driven by a one-fourth horsepower motor through a worm gear mechanism at a speed of four revolutions per minute. Heat is applied externally by gas burners. Two handholes, midway between the ends

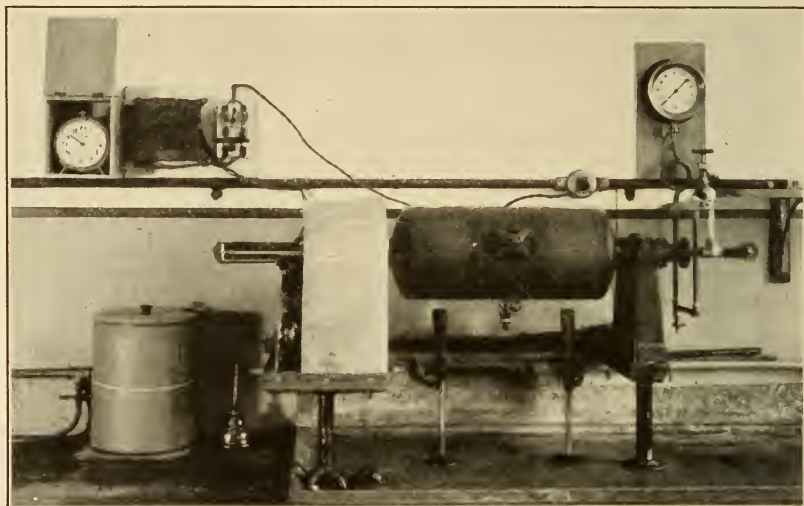


FIG. 1.—Laboratory rotary boiler

and opposite each other, permit easy removal of the cooked material. A thermometer, graduated in 5° units from 50 to 400° F., and a pressure gauge reading from 0 to 200 pounds are attached to the boiler.

(b) **BEATER.**—A one-half-pound beater (capacity one-half pound of dry fiber) is employed for the beating process necessary to produce suitable felting properties in the fibers.

(c) **FIBER SHEET MOLD.**—A method for making small sheets of fibers, uniform in quality (weight, formation, etc.) and free from the personal factor present in sheets made by hand by the usual method ⁵

⁵ In making hand sheets by the usual method, the diluted stock, contained in a vat, is thoroughly mixed and into it the hand mold is dipped. The deckle frame is filled and the mold is raised slowly, a shaking movement being meanwhile imparted to it by the operator. The water drains back into the vat thus changing the concentration of the remaining stock. Another sheet taken out at this stage would be of lighter weight. Therefore, it is customary to add to the vat, before taking out another sheet, stock equivalent to that removed by the preceding sheet. The possibility of error in the amount added and the personal factor in the shaking of the forming sheet tend to produce nonuniformity in the distribution of the fibers and in the weight of the sheets.

has been found essential for studying certain paper problems. The necessity for such sheets in the work of the paper laboratory has led to the construction by the writers of a fiber sheet mold and press which have given very satisfactory results.

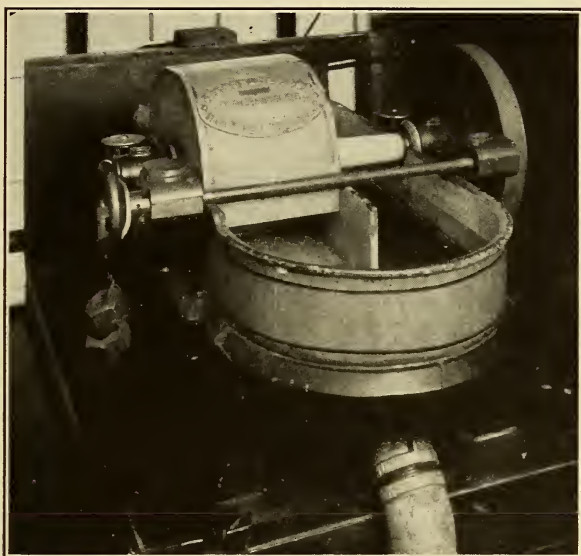


FIG. 2.—Laboratory beater

The mold consists of three castings, two brass and one aluminum (fig. 3, *A*, *B*, and *C*, respectively), a description of each of which follows:

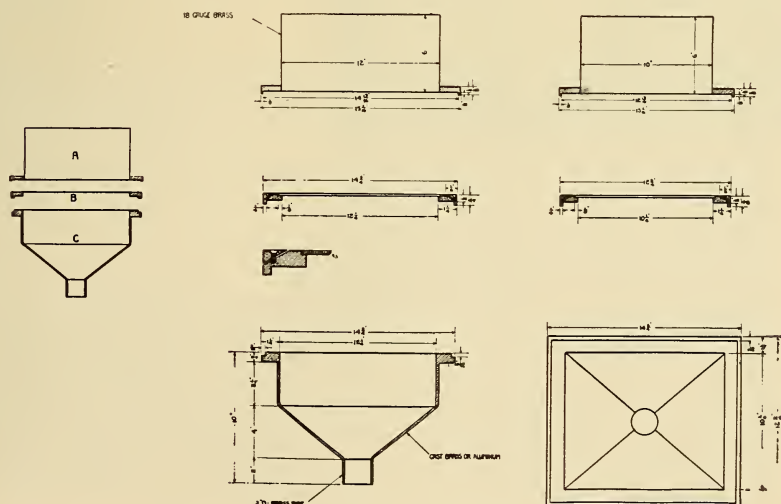


FIG. 3.—Working drawing of fiber sheet mold

Fitted into the base of the top section (*A*, fig. 3) is a sheet brass box, 10 by 12 inches, open at top and bottom and forming a deckle box.⁶ On the under side of the deckle base a rubber gasket is cemented (with shellac), thus making a tight seal when the deckle is clamped down (see fig. 4*A*). If there are many tests to be made it might be desirable to have the deckle box fastened to a swinging arm, so constructed that clamping is required at front only.

In the mold casting (*B*, fig. 3) is a perforated brass plate, *p*, drilled with $\frac{1}{2}$ -inch holes at $\frac{5}{8}$ -inch centers. A 10-mesh wire cloth is stretched over the plate and soldered to it at the edges. An 80-mesh wire cloth is stretched taut over all and the frame screwed down, thus holding the wire. Litharge and glycerine are poured on the sides to make the joint tight and prevent leaking.

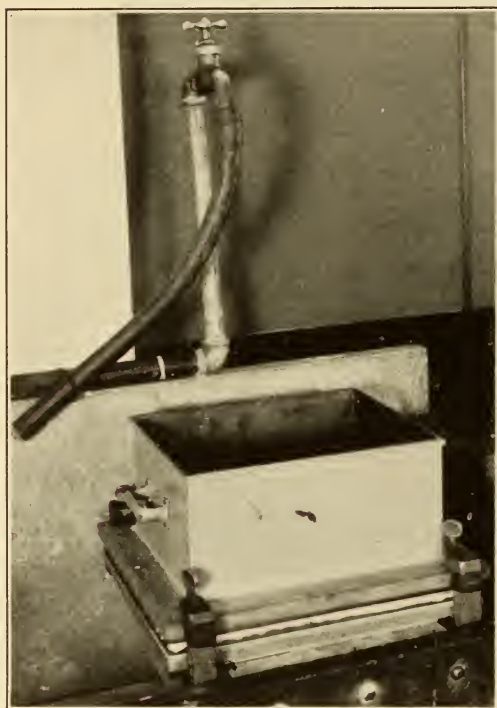


FIG. 4*A*.—Fiber sheet mold assembled

The mold casting in Figure 4*C* shows the results of this latter process. The screen plate, *p*, used to support the wire and prevent sagging, must be level if a uniform sheet is to be secured.

It may be noticed in Figure 4*C* that in the top of the lower casting (*C*, fig. 3) is a rubber tube fitted into a groove. This rubber supports the mold and

effects a tight seal. The outlet at the bottom of the lower casting is fitted with a quick-opening gate valve which, for suction purposes, is connected to a steam ejector below. The casting was made of aluminum, but brass could be used satisfactorily.

The quick-opening drain valve obtained for the outlet at the bottom was only $1\frac{1}{4}$ inches instead of 2 inches as the drawing of Figure 3 indicates. The valve used does not, therefore, permit as rapid emptying as would otherwise have been possible. The size of the opening provided for the discharge of the water should be kept in mind when considering subsequent data. (For comparison of rates of discharge see "Molding of test sheets," p. 332.)

⁶ A deckle box is a rectangular frame laid upon a wire mold to confine the paper pulp to a definite area.

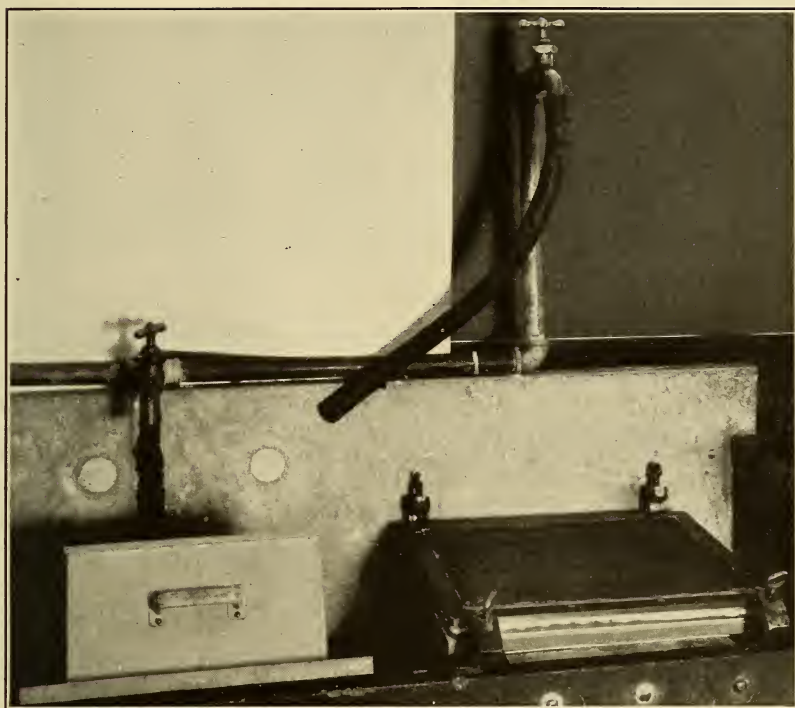


FIG. 4B.—*Fiber sheet mold with deckle box removed*

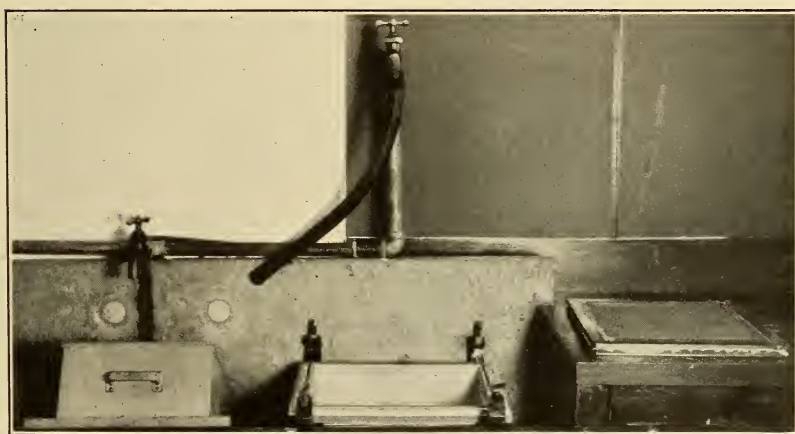


FIG. 4C.—*Fiber sheet mold with parts separated*

An interesting article by C. L. Batchelder, Forest Products Laboratory, United States Department of Agriculture, entitled "Pulp evaluation as affected by the fiber ratio in the test sheets,"⁷ states the following conclusions:

1. Fiber direction is the controlling factor in strength tests.
2. Before a sheet can be called uniform the fibers must be uniformly distributed. Such a condition can not be obtained when the hand mold is manipulated under usual methods.
3. The suction mold does eliminate many of the undesirable factors and produces a sheet more nearly perfect than can be made by other methods.

As indicated in these conclusions, care must be taken to prevent any differentiation in the arrangement of the fibers in a sheet; also, concentration of the stock used for the various sheets must be kept constant if the sheets are to be of similar quality. With the apparatus described above uniform distribution of the fibers is secured, and any sheet can be duplicated as frequently as desired.

Comparison of the measurements for the two directions of the sheet in the tensile strength, elongation, and tearing strength tests reported below shows that the fibers are uniformly distributed in sheets made on the sheet mold. The data of Table 1 were obtained for six different samples of stock. Ten sheets were tested for each sample, and the average of the 10 measurements is reported in the table.

TABLE 1.—Measurements for sheets from different samples of stock

Sample No.	Weight (25 by 40 inches— 500 sheets)	Bursting strength	Ratio bursting strength to weight (25 by 40—500)	Tensile strength (15 by 90 mm)		Elongation		Tearing strength	
				Long direc- tion	Short direc- tion	Long direc- tion	Short direc- tion	Long direc- tion	Short direc- tion
	<i>Pounds</i>	<i>Points</i>	<i>Per cent</i>	<i>kg</i>	<i>kg</i>	<i>Per cent</i>	<i>Per cent</i>	<i>g</i>	<i>g</i>
1.....	55.4	21.5	38.8	2.4	2.4	3.2	3.5	60.8	62.0
2.....	74.2	64.6	87.1	6.8	6.8	21.2	23.2	155.2	158.4
3.....	67.7	72.6	107.2	6.5	6.6	12.3	11.8	255.2	247.6
4.....	70.6	64.5	91.3	6.4	6.3	11.1	11.3	148.8	147.2
5.....	68.9	68.6	99.5	6.6	6.8	14.3	14.3	212.0	215.2
6.....	65.2	66.7	102.3	6.4	6.4	12.6	12.1	152.8	152.8

Table 2 shows that the sheet mold permits duplication of sheets. In this table are given for comparison the measurements on each of the 10 sheets from the stock of sample 1 above. The variations between different sheets are hardly greater than the usual experimental errors of measuring instruments and are far less than those commonly noted in sheets made on hand molds.

⁷ Paper, 27, No. 11, p. 14; November 17, 1920.

TABLE 2.—Measurements for 10 different sheets from one sample of stock

Bursting strength	Tensile strength (15 by 90 mm)		Elongation		Tearing strength (4 ply)	
	Long direction	Short direction	Long direction	Short direction	Long direction	Short direction
<i>Points</i>	<i>kg</i>	<i>kg</i>	<i>mm</i>	<i>mm</i>	<i>g</i>	<i>g</i>
20	2.7	2.0	2.50	3.00	16	16
21	3.1	2.3	3.25	3.00	16	14
23	2.0	2.1	2.75	3.25	17	16
20	2.2	2.3	2.25	1.75	16	15
22	2.4	2.3	3.00	3.50	15	17
23	2.3	2.7	2.50	3.50	15	17
21	2.6	2.4	3.75	3.25	14	16
21	1.8	2.3	3.00	3.50	15	17
20	2.9	3.1	3.00	3.50	15	14
21	2.0	2.5	3.00	3.25	13	15
¹ 21.2	¹ 2.40	¹ 2.40	¹ 2.90	¹ 3.15	¹ 15.2	¹ 15.5

¹ Average.

(d) SHEET PRESS.—Platform scales and a letter press were used in the construction of the sheet press (fig. 5). The upright supports for the upper part of the press are fastened to the base of the scales, but sufficiently distant from the sides of the platform to permit it to move freely. The sheets of fiber, between felts, are placed on the platform and the scales balanced. The top of the press is then screwed down, and the pressure is measured by the weights added to the lever-arm pan for balancing. Any pressure, up to the capacity of the platform scales, can be thus measured, kept uniform, and duplicated.

(e) DRYER.—Mechanical equipment was not available for drying the caroá paper, hence the pressed sheets were air-dried (in the open room). The small rotary steam-heated dryer shown in Figure 6 has since been installed and is now used for drying the sheets after their removal from the press.

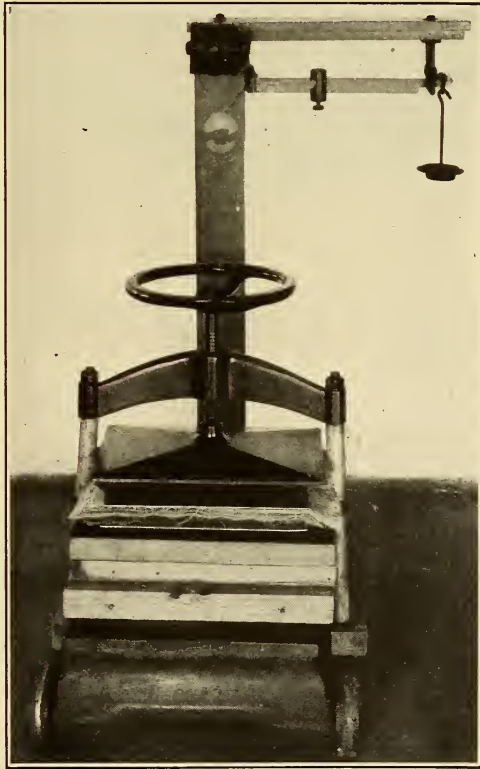


FIG. 5.—Sheet press

The dryer consists of a steam-heated cylinder and a carrying felt which covers most of the cylindrical surface. The diameter and the length of the cylinder are 12 and 14 inches, respectively. A steam-pressure gauge attached to the dryer assists in maintaining uniform temperature.

2. PROCEDURE

(a) BOILING OR "COOKING."—To facilitate the resolution of the plant materials by the action of the chemical solvents, the caroá was

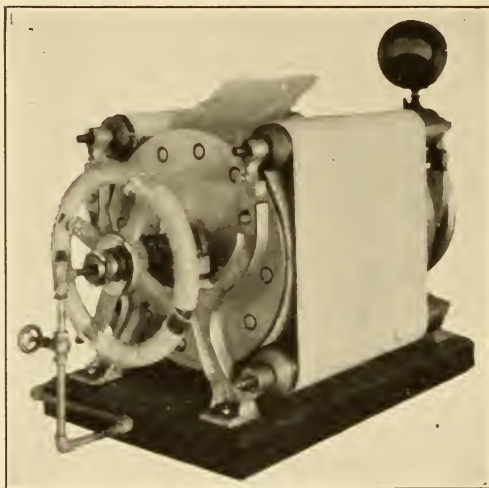


FIG. 6.—Laboratory dryer

cut into pieces about 1 inch in length, threshed, and dusted. Samples were taken for determination of moisture content. The material was then cooked in a solution of caustic soda (sodium hydroxide) in the small experimental rotary boiler (fig. 1). A number of cooks were made, keeping all conditions constant except the volume of caustic liquor, the percentage of caustic soda, based on weight of bone-dry material, being varied for each. The cooking constants were:

Weight of bone-dry material.....	grams..	908
Concentration of caustic liquor, in terms of Na_2CO_3 ⁸	grams/liter..	30
Causticity of cooking liquor.....	per cent..	9.98
Cooking temperature.....	° F..	342
Cooking pressure.....	pounds..	120
Time for raising to cooking temperature.....	hour..	1
Duration of cooking temperature.....	hours..	4
Time for cooling.....	hour..	½

(b) BEATING.—After being washed to eliminate the digested non-cellulose impurities and the cooking solution, the resultant material was bleached and beaten. A beater charge, "furnish," consisted of pulp equivalent to 135 g weight bone-dry, diluted to 1.75 per cent concentration. The beating consisted of only brushing out the fibers until there were no lumps or knots. No attempt was made to obtain maximum strength of fibers.

(c) MOLDING OF TEST SHEETS.—The beaten material was emptied into a copper pail and diluted with water until the weight of pulp and water was 10 kg (22 pounds), which weight gave a volume of

⁸ In commercial mills caustic soda cooking liquor is usually prepared from sodium carbonate. It is made by causticizing with quicklime.

⁹ 98 per cent of the soda was present as hydroxide

approximately 10 liters. The resultant stock was thoroughly mixed and a sample of 450 cm³ volume taken for each sheet. (A 450 cm³ sample gives a sheet of approximately standard basic weight, 60 pounds per 500 sheets, 25 by 40 inches in size—the exact weight depending on the shrinkage of the sheet.) The 450 cm³ sample was diluted still further in a 1-gallon copper measuring dish, the final concentration being approximately 0.16 per cent. The stock was then made into a sheet on the sheet mold.

The base, or mold support, fastened in a soapstone sink, was filled with water, and the mold and the deckle box were clamped in place (see fig. 4A). Water was then poured into the deckle box to a height of about 4 inches, making about 2 gallons of water above the wire, and the diluted stock in the 1-gallon copper measure was thoroughly agitated and poured into it, care being taken to distribute the stock evenly over the mold. The dilution at this stage is about 0.05 per cent, which is ample for good felting of fibers.

The steam-ejector valve was then opened and immediately after the quick-opening drain valve. The water and stock were drawn down, and the sheet of pulp was formed on the wire almost instantaneously. The time required for emptying depends on the "slowness" of the stock, but the time for emptying the machine when filled with water only is five seconds (for 1¼-inch pipe; for 2-inch pipe, two seconds should be sufficient). Quick emptying is necessary to prevent unequal distribution of fibers and material. It has been found desirable to continue the suction at least one minute after the base is emptied in order to hasten the drying of the sheet. After the sheet is formed the parts of the machine are separated, as shown in Figures 4B and 4C.

(d) PRESSING OF TEST SHEETS.—The sheet of pulp on the mold was covered with a napless felt, and over this a 4½-inch brass pipe (couch roll) was rolled to press out some of the water in the pulp and make easier the removal of the sheet from the wire. To insure uniform pressure on all sheets the weight of the pipe was the only pressure applied.

The sheet and felt, together, were then removed and placed felt down on a metal plate over which a similar felt had previously been laid. Another felt was placed over the sheet of pulp. The next sheet was similarly removed and placed on the upper felt of the first. This process was repeated until there were five sheets of pulp in the stack. With the addition of another felt and metal plate the unit was complete, and each sheet was provided with a double felt on either side. The felts and metal plates extended about 1 inch beyond the edges of the paper sheet.

The stack of alternating sheets and felts, with metal plates at top and bottom, was placed in the press (fig. 5), and a pressure of 900 pounds was applied for 10 minutes. (The pressure used was arbi-

trarily chosen. Additional work will be necessary to determine the pressure that yields best results.) The paper sheets were then removed and were ready for drying.

(e) DRYING TEST SHEETS.—The question of the proper method of drying is important. Work at the bureau has shown that paper dried on steam-heated rolls has lower strength than air-dried papers. Additional data will need to be obtained to determine the best method of drying.

As previously stated the drying process for the caroá papers did not include the use of the dryer equipment described on page 332. Instead, the pressed sheets of fiber were laid between sheets of wrapping paper and air-dried. The steam-heated dryer has been used, however, for the sheets made in subsequent tests on other fibrous materials. In using this equipment a sheet after being removed from the sheet press is fed between the dryer and felt, and the cylinder is turned slowly by hand. After two revolutions of the cylinder the sheet is removed, turned over, and replaced, care being used to avoid wrinkling the paper. The operation is repeated until the sheet is approximately bone-dry. The sheet is then trimmed to the desired size and later, immediately preceding the measurement of the physical properties, is conditioned similarly to all other paper samples in a constant temperature and constant humidity room (temperature 70° F.; relative humidity 65 per cent).

3. TEST RESULTS

(a) LOSSES IN CLEANING.—The loss of weight in the initial operations of cutting, threshing, and dusting the caroá material was: Sample A, 3.5 per cent; B, 6.0 per cent; and P, 7.1 per cent.

(b) BASIS FOR PERCENTAGES.—The values in the first four columns of percentage data in Table 3 are based on the weight of the bone-dry material after cutting, threshing, and dusting.

(c) TABULAR DATA ON PAPER MADE.—The first part of Table 3 gives data on caustic soda (in terms of Na_2CO_3) added and consumed,¹⁰ yield of pulp, and bleaching qualities. Under "Bleach required" is given the amount of bleaching powder (calcium hypochlorite), containing 35 per cent of available chlorine, required for bleaching to a good color. The values under "Physical tests on finished paper" have been converted for comparative purposes to a 60-pound weight basis.

¹⁰ For those not familiar with a suitable method for determining consumption of soda, the following formula ("Chemistry of Pulp and Paper Making," by Edwin Sutermeister, S. B., p. 110; 1920) is given and is a condensed statement of the procedure employed in the tests reported above.

"If the causticity of the liquor as added is known, the consumption of caustic soda may be calculated at any time from the black-liquor analysis by means of the following formula:

$$X = C - \frac{B \times C}{A}$$

"where

"X=per cent NaOH used up, based on the bone-dry material,

"A=per cent causticity at the start,

"B=per cent causticity at time of sampling black liquor,

"C=per cent NaOH added on bone-dry material."

TABLE 3.—Data on finished paper of laboratory tests

Caroá sample	Test No.	Caustic soda added (on bone-dry basis)	Caustic soda consumed	Yield of pulp	Bleach required	Physical tests on finished paper								
						Weight (25 by 40 inches—300 sheets)	Bursting strength	Ratio bursting strength to weight (25 by 40—500)	Tensile strength (strip 15 by 90 mm)		Elongation		Tearing strength	
									Long direction	Short direction	Long direction	Short direction	Long direction	Short direction
		Per cent	Per cent	Per cent	Per cent	Pounds	Points	Per cent	kg	kg	Per cent	Per cent	g	g
A	1	20.0	20.0	43.7	(2)	60.0	73.2	122.0	6.2	6.3	14.4	16.6	216.3	224.0
A	2	27.7	26.2	39.1	Over 13	60.0	52.2	87.0	5.5	5.5	17.1	18.7	125.8	128.0
B	3	15.0	15.0	55.5	(2)	60.0	85.3	142.1	7.3	7.9	14.3	16.0	207.0	219.0
B	4	20.0	19.7	49.3	13	60.0	59.8	99.7	5.7	5.9	12.5	12.5	185.0	187.2
B	5	25.0	23.2	49.3	13	60.0	61.4	102.3	5.9	5.9	11.6	11.1	140.5	140.5
B	3	25.0	23.5	45.1	13	60.0	54.3	90.5	5.2	5.4	14.6	14.7	126.2	126.2
P	7	10.0	10.0	67.0	(2)	60.0	64.4	107.3	5.7	5.8	10.9	10.5	219.4	219.4
P	8	15.0	15.0	61.2	(2)	60.0	63.9	106.5	6.5	6.3	9.4	8.8	209.0	213.0
P	9	20.0	19.4	60.0	13	60.0	59.5	99.1	6.3	6.3	9.8	9.5	140.7	146.2
P	10	21.9	20.4	59.7	13	60.0	54.8	91.3	5.4	5.4	9.4	9.6	126.2	125.1
P	11	25.0	21.0	55.3	10	60.0	52.1	86.8	5.2	5.5	10.4	10.8	111.5	116.2
P	4	11a				60.0	50.3	83.8	5.1	5.3	9.8	10.4	112.8	109.5
Sulphite ⁵						60.0	23.3	38.8	2.6	2.6	3.4	3.8	65.8	67.2

¹ Sample taken from sheet (as formed on the sheet mold) in direction indicated.² Pulp remained unbleached even when amount of bleach added exceeded 16 per cent and was therefore regarded as commercially unbleachable.³ Sample B, test No. 6, was cut only; not threshed or dusted.⁴ Test 11a was from the same pulp as test 11 and was made as a check test.⁵ A test on sulphite pulp was included as a basis of comparison.

The data show, as would be expected, the yield of pulp highest for the samples previously treated (purified) most; also, less caustic required to reduce these samples. The proportion of caustic used was a big factor in determining the yield as well as the bleaching properties of the fiber.

Sample A, which had received no preliminary treatment (being in its original state except for shredding), contained a large amount of pithy material. It, therefore, required more caustic soda for reduction to a bleachable pulp than the treated samples and gave a proportionately lower yield. The yield for test No. 1, using 20 per cent of caustic soda, was 43.7 per cent and the pulp was unbleachable, but the paper made from it showed unusual strength. This was due not only to the strength of the fibers themselves but also to the cementing action of the unremoved pithy material. The pulp was very hard to wash, and it beat to a "slow" stock in a very short time. There would be no objection to leaving a small amount of this pithy material in the stock when making bag or wrapping papers. In these papers strength is required, and this material would tend to bind the fibers and give a certain amount of waterproofing. If much were left, however, the trouble in washing and beating and the decrease in speed of production would probably be greater than the advantage gained would warrant. Test No. 2 gave a cleaner fiber, but the yield was low.

Sample B had received some preliminary mechanical treatment and, therefore, gave an increased yield. When 25 per cent of caustic soda was used, the pulp bleached (with 13 per cent of bleach) to a very good white and would have been satisfactory for use in high-grade papers, such as book and writing.

Sample P had lost much of its pithy material in the preliminary fermentation treatment. The yield was very good even when pulp that bleached easily was obtained. The bursting strength (and consequently the ratio of bursting strength to weight), decreased of course, with increase in amount of caustic added, but the paper from the weakest stock tested nearly a point per pound. The tensile strength showed practically no difference with increasing amounts of caustic, but the tearing strength dropped 50 per cent from the first (10 per cent of caustic) to the last (25 per cent of caustic). With the lower amounts of caustic, pulps were obtained which made excellent paper well suited for bag or wrapping; with the higher amounts, the pulp obtained compared favorably with rag stock. The stock of test No. 11 washed easily and bleached to a white with 10 per cent of bleach. The pulp handled well in the beater, brushing out with very little trouble, and gave a very well-formed sheet on the sheet mold. The physical tests showed the finished paper to be very strong. A check beater test was made, and the results are given in test No. 11a.

It has been reported ¹¹ that materials similar to caroá fiber have been treated by a bacterial process with the result that very little bleach is required to obtain from the resultant product a white stock suitable for paper manufacture. If this process would work satisfactorily on caroá fiber, it might be advisable to apply it at the time of harvesting. The material would then be in such condition when shipped that paper could be made from it without any further treatment than that ordinarily given rag or rope stock. No data have been obtained as to the cost of the bacterial process or the subsequent yield of paper-making fiber.

The yield of paper-making fiber from caroá compares very favorably with that for rags (60 to 85 per cent), hemp (50 to 65 per cent), or jute (50 to 65 per cent).¹² The bursting strength of paper made from caroá is about a point per pound (at standard basic weight, 60 pounds per 500 sheets 25 by 40 inches), which is the strength specified for the best papers made from rag or rope.

IV. SEMICOMMERCIAL PAPER-MAKING TESTS

The laboratory tests have indicated that caroá fiber compares favorably with materials commonly used for the manufacture of paper, but for commercial interpretation and comparison results obtained by practical mill tests also are desired. Therefore, tests were also made in the Bureau of Standards experimental paper mill and are described in the following.¹³

¹¹ See footnote 2, p. 325.

¹² The Manufacture of Pulp and Paper, 4, pp. 38, 41, 44.

¹³ A brief description of commercial paper-making processes is included for the reader not familiar with paper manufacture. Paper making consists of two stages, namely, the preparation of the pulp and the conversion of the prepared pulp into a dry continuous sheet of paper. The plant material from which the pulp is obtained is freed from foreign matter, such as sand and dirt, and reduced to pieces of suitable size by the preliminary operations of dusting and cutting. The action of chemical solvents in the boiling or "cooking" process which follows dissolves or separates the noncellulose constituents, which are impurities in paper-making stock, from the cellulose fiber, which is the basis of all papers. After the soluble compounds formed are removed by washing, subsequent bleaching with oxidizing agents, followed by washing again, completes the chemical cleansing of the pulpy residue.

The paper-making fibers are reduced to optimum length, brushed out, and frayed in the beater (see fig. 9), an oblong tub fitted with a revolving roll containing metallic blades which engage with the bars of a metallic plate at the bottom. After the beating has been carried on sufficiently long to prepare the fibers for effective interlacing in the final processes, the beater contents are discharged into a storage tank, the beater chest. From the beater chest the stock is pumped in a continuous stream to the stuff box where the rate of flow to the Jordan is regulated. The Jordan (fig. 10), a refining engine used to give the fibers a final brushing out so that they will flow independently in the watery medium, consists of a conical-shaped plug equipped with metallic bars and rotating inside a similarly shaped shell, likewise equipped with metallic bars. After passing through the Jordan the diluted stock flows through a screen, which permits only individual fibers to pass through, to the Fourdrinier machine where the paper is formed (fig. 11, A and B).

The stock flows onto a traveling endless wire cloth which is given a sidewise reciprocating motion to assist the fibers to interweave in all directions. The bulk of the water drains through the wire cloth as the sheet formed is carried forward. As much as possible of the remaining water is removed by passing the sheet over suction boxes, then successively under a felt-covered couch roll, where the sheet is transferred to an endless woolen felt, between press rolls which remove more water, and over large hollow steam-heated drying cylinders. The paper passes next through a stack of highly polished metal calendering rolls which impart a polish to the sheet, and then to the reel on which the finished paper is wound.

1. EQUIPMENT

The paper mill of the bureau is equipped for making paper on a semicommercial scale under practical mill conditions. The equipment employed in the caroá investigation was that in general use in the bureau mill and consisted of a rag duster, rag cutter, rotary boiler, 50-pound copper-lined wood tub beater (with manganese-bronze bars and plate) equipped with a washing cylinder, small Jordan (with iron bars), a four-plate screen, and a 29-inch Fourdrinier paper-making machine (with wire 33 feet long and having two presses, nine 15-inch dryers, a small machine stack of seven rolls, and a reel).



FIG. 7.—Duster and cutter

Figures 7 to 11B are photographs of this equipment. The ampere-meter recorders shown in Figures 9 and 10 enable close control of the beating and jordanning processes. Watt-meter recorders, however, would doubtless be preferable to ampere-meter recorders in the installation of new equipment.

2. PROCEDURE

The procedure followed in the mill tests was essentially the same as that generally observed in the commercial production of high-grade papers.

In the preliminary treatment the caroá material, which was received in the form of rope, was untwisted, pulled apart, and put through the duster. The action of the duster separated and threshed

the material and eliminated much of the dust and other foreign matter, such as hard, dark-colored particles, which would not have been removed in the pulping processes and would have caused dark specks in the finished paper.

The material was cut into pieces approximately 1 inch in length and charged into the boiler. A number of cooks were made, the variations in which are shown in Table 4. The cooking constants were:

Causticity of cooking liquor (caustic cooks).....	per cent.....	98
Cooking temperature.....	°F.....	300 to 310
Time for raising temperature.....	hour.....	1
Duration of cooking temperature.....	hours.....	6
Cooking pressure.....	pounds.....	65 to 75

The pulp from which writing papers were to be made was bleached, a solution of calcium hypochlorite being the oxidizing agent used.

In the paper-making process the stock was jordanned directly to the paper machine.

3. TEST RESULTS

(a) LOSSES IN CLEANING.—The loss of material in threshing and cutting was less than 2 per cent.

(b) BASIS FOR PERCENTAGES.—The yield of pulp and other percentage values are based on the bone-dry weight of the material after threshing and cutting.

(c) TABULAR DATA ON PAPER MADE.—Numerical data relative to the chemical reduction of the caroá material and measurements on

the finished paper are given in Table 4. Similar measurements for high-grade rag and sulphite writing papers and kraft wrapping paper are included for comparison, but inasmuch as the tests on these papers were made previous to the present investigation some of the corresponding values could not be supplied.

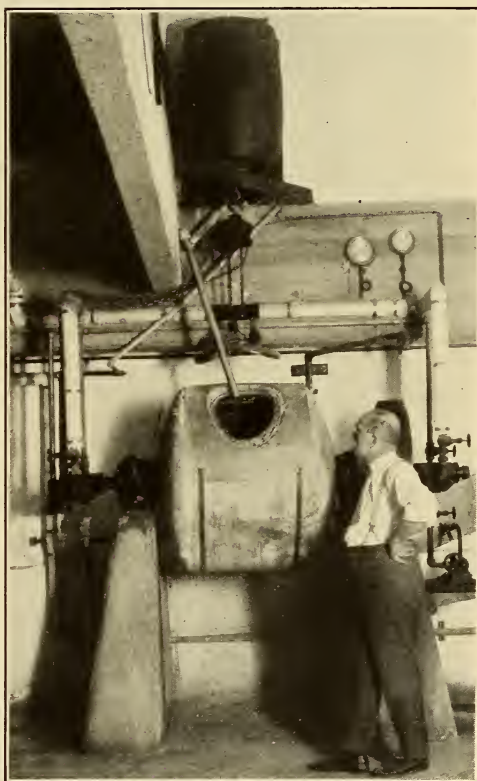


FIG. 8.—Rotary boiler

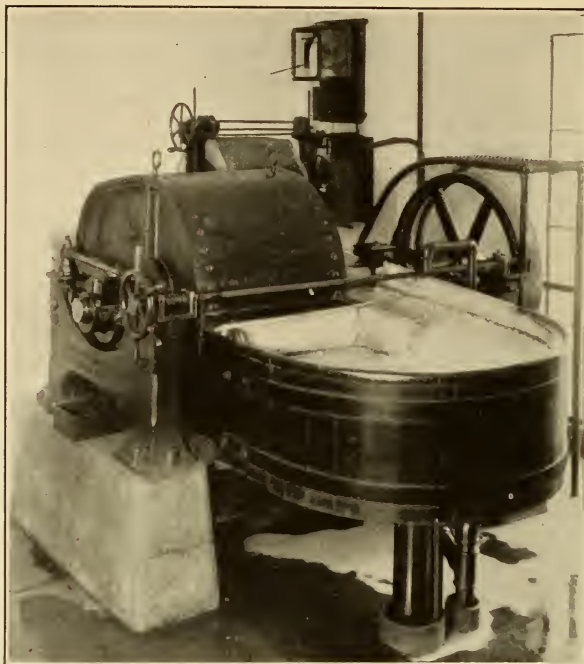


FIG. 9.—*Beater*
Ammeter chart for motor indicated by arrow

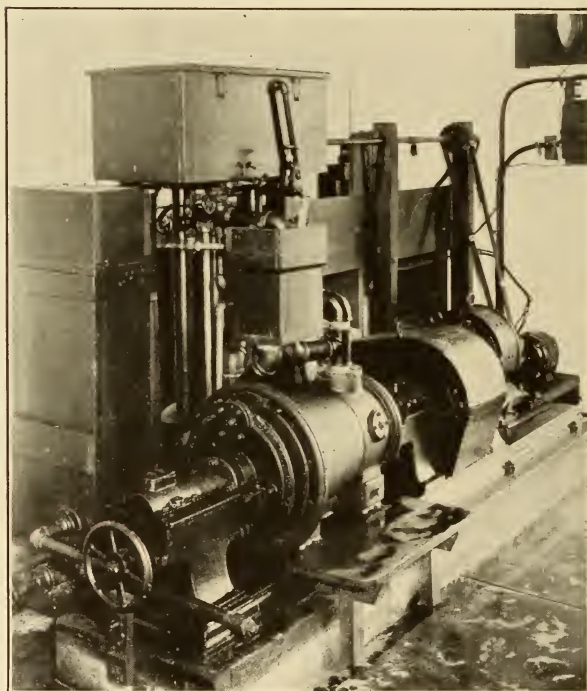


FIG. 10.—*Jordan*
Ammeter chart for motor indicated by arrow

Machine runs Nos. 620 and 621 were wrapping paper, therefore no attempt was made to bleach the pulp for these runs. The digesting materials employed in the corresponding cooks were caustic soda (sodium hydroxide) and lime (calcium oxide), respectively.

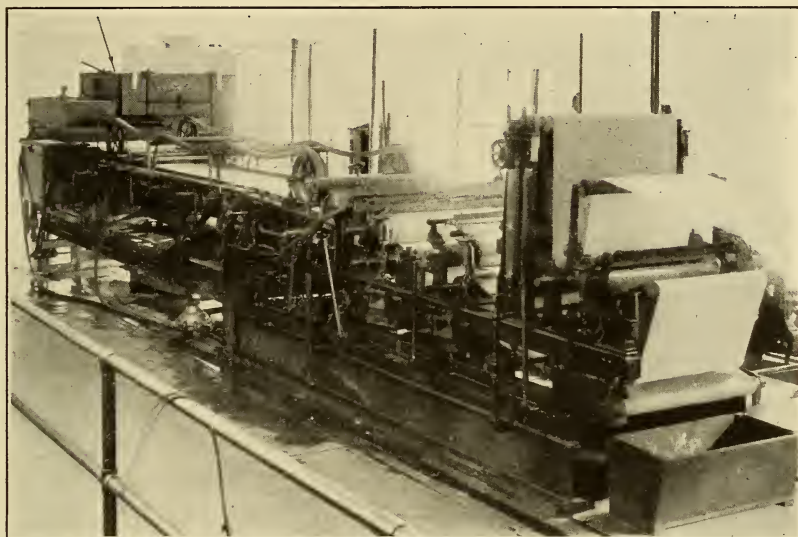


FIG. 11A.—*Fourdrinier machine*
Four-plate screen indicated by arrow

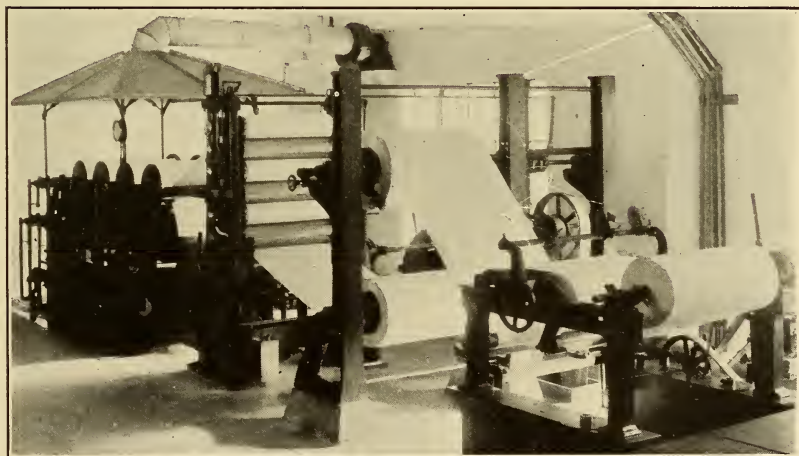


FIG. 11B.—*Fourdrinier machine*

The measurements for the finished papers are on air-conditioned test samples (65 per cent relative humidity and 70° F. temperature). The test methods employed conform to the official paper-testing methods of the Technical Association of the Pulp and Paper Industry, with whom this bureau collaborated in their preparation.

TABLE 4.—Data on finished paper of semicommercial tests

Physical tests on finished paper										Sizing tests ¹		Chemical tests					
Machine run No. 1	Caustic soda added (on bone-dry basis)	Yield of pulp	Bleach required	Weight by 40 inches—500 sheets)	Bursting strength	Thickness	Ratio bursting strength to weight (25 by 40—500)	Tensile strength (15 by 90 mm)		Tearing strength		Folding endurance		Indicator method	Curl method	Ash	Resin (alcohol method)
								Machine direction	Across machine direction	Machine direction	Across machine direction	Machine direction	Across machine direction				
Per cent	Per cent	Per cent	Per cent	Pounds	Points	Inch	Per cent	kg	kg	g	g	Double folds	Double folds	Seconds	Seconds	Per cent	Per cent
616A.....	27.1	21.6	51.5	8.0	55.3	58.9	0.0040	110.4	9.3	5.9	107.6	108.4	2,235	2,485	19.1	1.52	1.10
616B.....	27.1	21.6	51.5	8.0	59.6	52.4	.0043	87.9	9.5	6.3	119.6	126.4	2,095	2,695	20.3	1.84	2.68
618.....	25.0	22.1	49.5	10.0	57.2	61.1	.0044	106.9	9.2	6.7	108.8	112.0	1,845	2,975	9.6	1.98	2.10
619.....	25.0	20.8	46.8	10.0	56.3	59.0	.0043	104.8	10.8	6.3	126.8	125.2	3,350	3,250	17.8	1.87	2.35
617.....	23.1	21.0	48.5	10.0	55.5	63.1	.0043	113.7	11.1	7.3	118.4	126.0	3,045	3,895	12.7	1.96	2.88
620.....	12.5	12.5	57.5	(°)	52.2	61.0	.0040	116.8	8.9	5.6	146.0	141.0	3,250	4,165	20.7	1.17	3.75
621.....	(°)	-----	60.4	(°)	52.0	52.3	.0041	100.5	9.6	5.2	138.4	123.2	2,915	2,655	19.9	2.13	3.15
Rag (writing) ²	-----	-----	-----	-----	63.0	38.5	.0041	61.1	8.2	4.0	152.0	152.8	3,355	2,055	-----	.70	.80
Sulphite (writing) ³	-----	-----	-----	-----	54.3	32.2	.0037	59.3	-----	-----	61.4	51.0	855	545	22.6	.22	2.02
Kraft (wrapping) ⁴	-----	-----	-----	-----	55.8	55.1	.0041	98.7	-----	-----	106.8	92.5	3,428	3,045	19.7	.75	1.07

¹ For papers of the thickness of these samples, values of 18 to 20 seconds indicate a well-sized sheet.² Waterleaf.³ Wrapping paper; no attempt made to bleach.⁴ No soda; 25 per cent lime.⁵ Measurements on rag, sulphite, and kraft pulps are included as a basis for comparison.

Sample B of the laboratory tests and the caroá material employed in the semicommercial work had undergone similar processes in the preliminary separation of the fiber from the plant, therefore comparison of the test results for these materials may be made. Comparing the tests in which equal amounts of caustic soda were added—No. 5, Table 3, and machine runs Nos. 618 and 619, Table 4—it is evident that the results of the paper-mill work check closely with those obtained in the laboratory study. The percentage of caustic soda consumed, the yield of pulp obtained, and the ratio of bursting

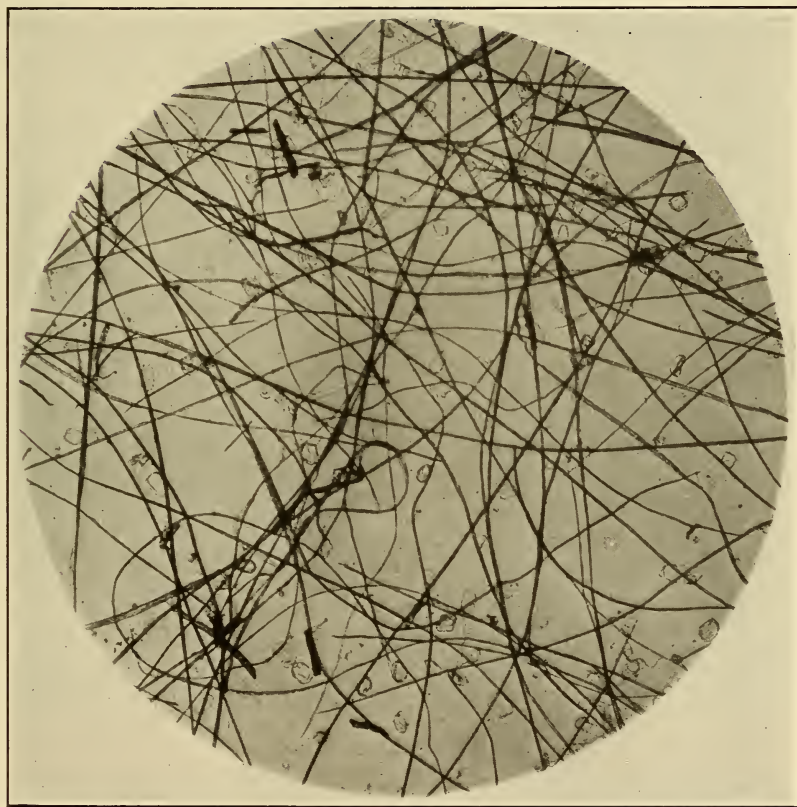


FIG. 12.—Caroá fiber

strength to weight are practically the same in the laboratory and the semicommercial tests. The greater tearing strength for No. 5, Table 3, is due in part to the greater weight of the test sample and the fact that the hand sheets were air-dried, whereas the paper-machine product was dried by steam-heated cylinders.

V. FIBER CHARACTERISTICS

Figures 12, 13, and 14 are photomicrographs¹⁴ of caroá fiber, caroá and rag fibers, caroá and sulphite fibers, respectively. The

¹⁴ Photomicrographs and caroá fiber measurements were made by R. E. Lofton, Bureau of Standards; dimensions of other fibers were taken from standard textbooks.

photomicrographs were made at a magnification of 100 diameters, but for the reproductions herein the magnification is about 55. Figure 12 shows the caroá fiber to be long, of small diameter, and cylindrical with pointed ends. These properties produce good felting of the fibers and give compactness and strength to the resulting sheet.

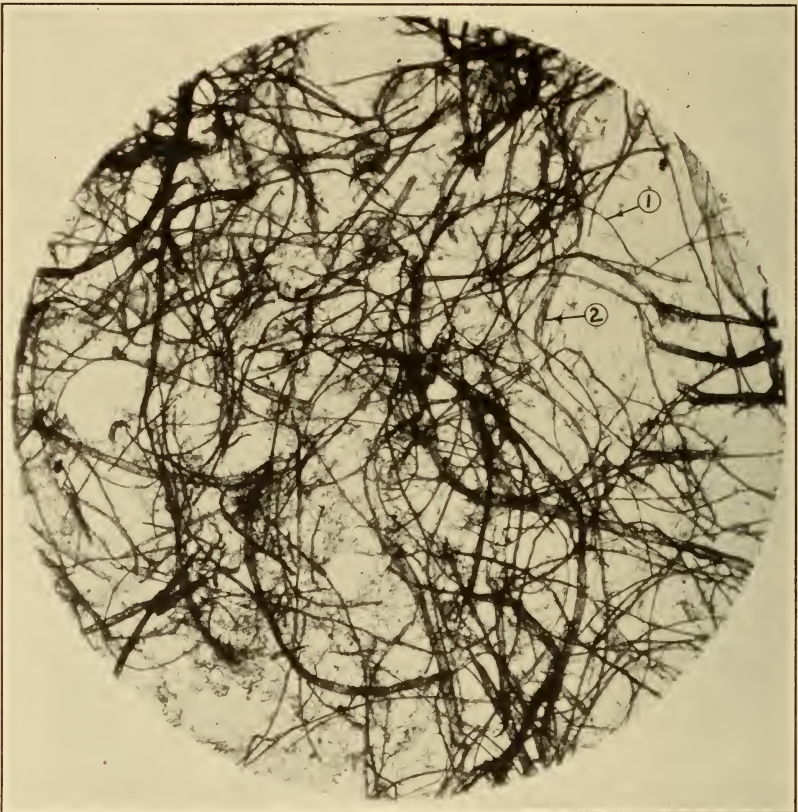


FIG. 13.—Caroá and rag fibers
Fibers are indicated by ① and ②, respectively

The following fiber measurements¹⁵ also afford comparison between caroá and other paper-making materials:

Fiber	Length	Diameter	Ratio of length of diameter
	<i>mm</i>	<i>mm</i>	
Cotton.....	20-50	0.012-0.037	1400 : 1
Flax.....	25-30	“ .02	1200 : 1
Manila hemp.....	“ 6	“ .024	250 : 1
Jute.....	“ 2	“ .022	90 : 1
Sulphite pulp.....	“ 2.8	“ .033	90 : 1
Caroá.....	“ 4.0	“ .010	400 : 1

^a Average.

¹⁵ See footnote 14, p. 343.

The range of the dimensions of the caroá fiber was: Diameter, 0.0046 to 0.0154 mm; length, 2.25 to 5.75 mm. Although cotton and linen fibers have better ratio of length to diameter, they are much too long for paper making and have to be reduced to about the same length as caroá fiber. The latter should have better felting quality because of its smaller diameter.

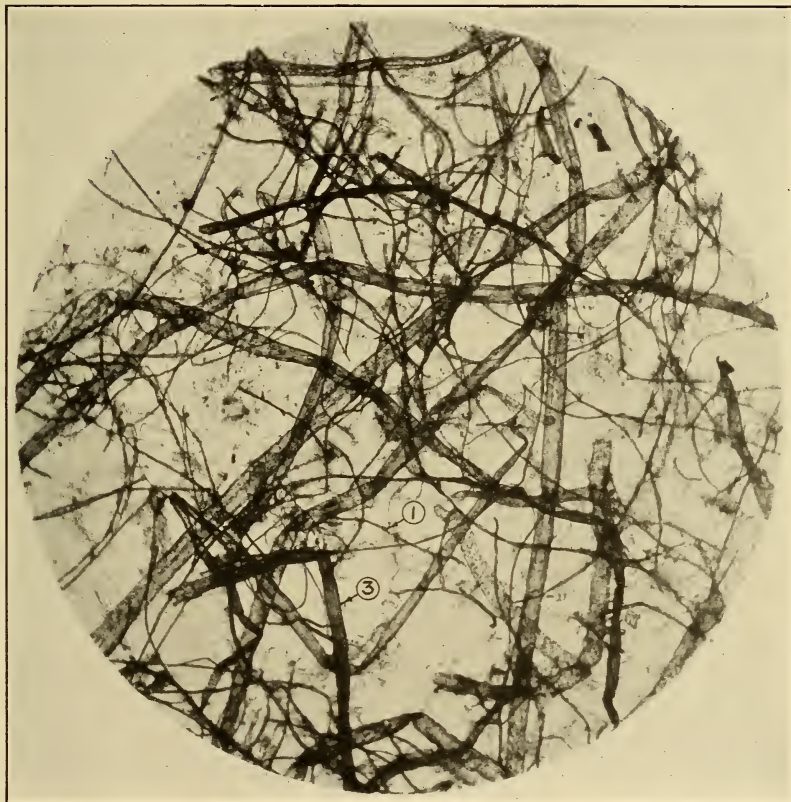


FIG. 14.—*Caroá and sulphite fibers*
Fibers are indicated by ① and ③, respectively

VI. CONCLUSIONS

Laboratory and semicommercial tests have shown the caroá plant to be a very satisfactory paper-making material, on the basis of both quality of fiber produced and yield of fiber obtained.

The preliminary process employed in the separation of the fiber from the plant is a big factor in determining the properties of pulp made from caroá. For the fibrous material separated by mechanical processes the yield is low and bleaching the pulp is not practical, but the paper made therefrom is very strong. Material obtained by retting gives a good yield of pulp which bleaches easily and pro-

duces a strong paper, although of less strength than that made from the fiber obtained by mechanical separation only.

When cooked by the caustic-soda process, with the smaller amounts of caustic, caroá produces paper which is excellent for bag or wrapping purposes and is as strong as that made from kraft pulp; with the larger amounts of caustic, the pulp is stronger than sulphite and compares favorably with high-grade rag stock.

A micrographic study of caroá shows the fiber to be cylindrical and comparatively long and of small diameter. These properties tend to produce good felting of fibers and give compactness and strength to the resulting sheet.¹⁶

Determination of the suitability of a material for paper making includes, in addition to the evaluation of the product made from it, the ascertaining of the potential production and concentration of a sufficient supply of the raw material. Estimates relative to the potential production and cost of caroá as a raw material of commerce are somewhat meager, but the economic availability of the plant appears promising.

¹⁶ It is interesting to note here the following statement from the abstract of an article on caroá fiber (published complete in "*L'Industria della carta*," **28**, 15; 1925) given in *Papierfabrikant*, **23**, No. 24, p. 391; June, 1925. "It is concluded from the chemical, mechanical, and microscopic tests of the fiber, as well as from the tests of the sample of paper, that, after perfecting the process for separating the fiber and for bleaching, we shall have a raw material that is eminently suited for the preparation of paper."

WASHINGTON, January 29, 1927.



